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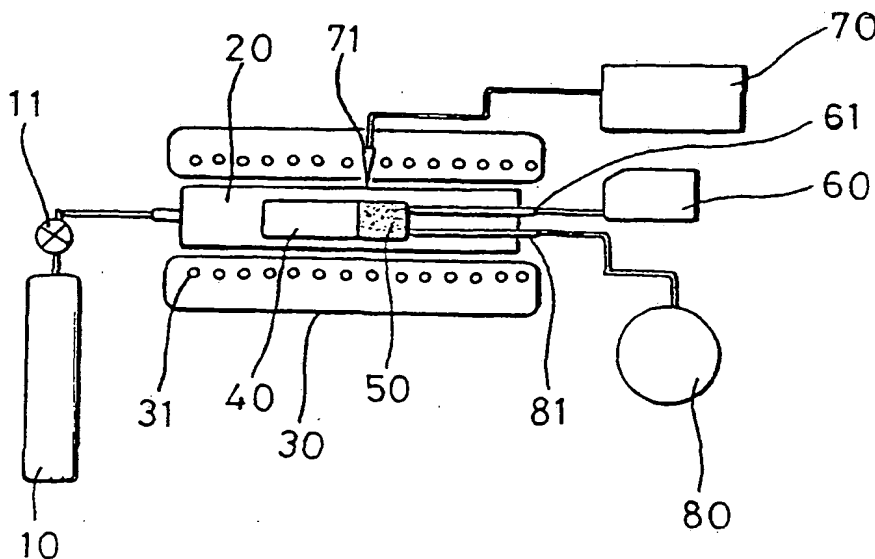
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(54) Title: APPARATUS AND METHOD FOR MANUFACTURING AN ARTIFICIAL POROUS TITANIUM NICKEL MEDULLA BY USING A HOT ROTATIONAL SYNTHESIS METHOD



(57) Abstract: An apparatus and method for manufacturing an artificial porous titanium nickel medulla by using a hot rotational synthesis method is disclosed. The method includes the steps of drying raw powders of titanium and nickel under the vacuum state, dry-mixing the raw powders with each other at a ratio of about 1:1, molding the mixed powders within a cylindrical quartz tube with compression or without pressure, reacting the mixed powders molded in the molding step in a reaction furnace by a hot rotational synthesis method, cooling titanium-nickel products reacted in the reacting step using a reservoir for a cooling liquid, and removing impurities on a surface of the cooled titanium-nickel products to process the titanium-nickel products in a desired shape.

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APPARATUS AND METHOD FOR MANUFACTURING
AN ARTIFICIAL POROUS TITANIUM NICKEL MEDULLA
BY USING A HOT ROTATIONAL SYNTHESIS METHOD

5 Techincal Field

The present invention relates to an apparatus and method for manufacturing an artificial porous titanium-nickel medulla by using a hot rotational synthesis method, and more particularly to an apparatus and method for
10 manufacturing an artificial porous titanium nickel medulla by using a hot rotational synthesis method, in which a porous structure is controlled to obtain mechanical qualities required for an implant material at a site of a shattered medulla which absolutely requires
15 initial stability, thereby enhancing osteoconduction effect and bone ingrowth, and a newly grown medulla is created after implant and then its material qualities are controlled to be similar to a human medulla, thereby obtaining biocompatibilty.

20

Background Art

In general, after lesion of a patient having a shattered medulla, such as a joint defect, trauma, and medulla tumor, is excised, automedulla grafting,
25 isomedulla grafting or heteromedulla grafting is operated to protect legs and arms of the patient.

However, such medulla grafting such as automedulla grafting, isomedulla grafting and heteromedulla grafting has several problems. That is, the medulla grafting is subject to various limitations as to whether bio-

5 compatibility exists or not depending on both the site of the shattered medulla and body qualities of the patient.

Meanwhile, in the prior art, an artificial medulla for grafting on a site of a shattered medulla is manufactured based on powder metallurgy method. In the

10 powder metallurgy method, the artificial medulla is manufactured in such a manner that the site of the shattered medulla of a patient is incised and then metal or ceramics powder is compressed and sintered to protect

15 legs and arms of the patient without subjecting the patient to factors such as the site of the shattered medulla and body qualities.

However, the power metallurgy method has several problems. That is, the power metallurgy method does not permit a porous structure having mechanical qualities

20 suitable for the site of the shattered medulla of the patient to be manufactured. Also, the artificial medulla is not controlled to be similar to an actual medulla of a human body, thereby causing some problems in biocompatibility.

Disclosure Of Invention

Accordingly, an object of the present invention is to solve at least the problems and disadvantages of the prior art.

5 Another object of the present invention is to provide an apparatus and method for manufacturing an artificial porous titanium nickel medulla by using a hot rotational synthesis method, in which a porous structure is controlled to obtain mechanical qualities required for
10 an implant material at a site of a shattered medulla which absolutely requires initial stability, thereby enhancing osteoconduction effect and bone ingrowth.

Other object of the present invention is to provide an apparatus and method for manufacturing an artificial
15 porous titanium nickel medulla by using a hot rotational synthesis method, in which a newly grown medulla is created after implant and the its material qualities are controlled to be similar to a human medulla, thereby obtaining biocompatibility.

20 To achieve the objects and in accordance with the purposes of the invention, as embodied and broadly described herein, a method for manufacturing an artificial porous titanium nickel medulla by using a hot rotational synthesis method according to the present
25 invention includes the steps of drying raw powders of titanium and nickel under the vacuum state to remove

moisture and surface absorption materials, thereby
minimizing an amount of gas generated during synthesis
reaction, dry-mixing the raw powders dried during the
drying step with each other at a ratio of about 1:1 in
5 atomic amount to manufacture mixed powders having uniform
compositions, molding the mixed powders within a
cylindrical quartz tube with compression or without
pressure depending on a desired porosity and pore size,
reacting the mixed powders molded in the molding step in
10 a reaction furnace by a hot rotational synthesis method,
cooling titanium-nickel products reacted in the reacting
step using a reservoir for a cooling liquid, and removing
impurities on a surface of the cooled Titanium-nickel
products to process the titanium-nickel products in a
15 desired shape.

Brief Description Of Drawings

The invention will be described in detail with
reference to the following drawings in which like
20 reference numerals refer to like elements wherein:

Fig. 1 is a view showing the manufacturing steps of
an artificial medulla according to the present invention;
and

Fig. 2 is a view showing an apparatus for
25 manufacturing an artificial medulla according to the
present invention.

Best Mode for Carrying out the Invention

Reference will now be made in detail to the preferred embodiments of the present invention, examples of which are illustrated in the accompanying drawings.

5 A method for manufacturing an artificial porous titanium nickel medulla by using a hot rotational synthesis method according to the present invention includes the steps of drying raw powders of titanium and nickel under the vacuum state to remove moisture and
10 surface absorption materials, thereby minimizing an amount of gas generated during synthesis reaction, dry-mixing the raw powders dried during the drying step with each other at a ratio of about 1:1 in atomic amount to manufacture mixed powders having uniform compositions,
15 molding the mixed powders within a cylindrical quartz tube with compression or without pressure depending on a desired porosity and pore size, reacting the mixed powders molded in the molding step in a reaction furnace by a hot rotational synthesis method, cooling titanium-
20 nickel products reacted in the reacting step using a reservoir for a cooling liquid, and removing impurities on a surface of the cooled titanium-nickel products to process the titanium-nickel products in a desired shape.

Preferably, in the drying step, the raw powders of
25 titanium and nickel are dried for 8 hours or more at a temperature between 60°C and 70°C under the vacuum state.

Also, in the mixing step, the dried powders are dry-mixed with each other by a ball mill for 10 ~ 12 hours.

Meanwhile, when dry-mixing the powders, a vessel
5 such as glass material is used to prevent the mixed powders from being contaminated. However, it is preferable that a grinder such as a steel mill is not used.

Furthermore, it is preferable that a molding body
10 molded within the quartz tube has a diameter of 20mm or greater to avoid instability (extinguishment) of the ignition wave due to heat loss and to obtain adiabatic reaction conditions.

It is also preferable that the molding body has a
15 porosity of 30 ~ 70%. If porosity is too low, heat loss caused by heat transfer during ignition reaction increases. This could prevent the ignition wave from being propagated. That is, it is likely that the ignition wave is extinguished. On the other hand, if porosity is
20 too high, intensity becomes low so as not to be handled in the manufacturing steps.

Grinding or turning removes surface impurities of the cooled titanium-nickel products so that the discharge processing processes the cooled titanium-nickel products
25 step in a desired shape. It is noted that the surface impurities are pickled with a mixed solution of distilled

water of hydrofluoric acid and nitric acid, so as to be removed.

Meanwhile, as shown in Fig. 2, an apparatus for manufacturing an artificial porous titanium nickel medulla by using a hot rotational synthesis method according to the present invention includes an inert gas receptacle 10 for containing an inert gas of which amount is controlled by a gas flow controller 11, a tubular reactor 20 to which the inert gas contained in the inert gas receptacle 10 is supplied, a titanium-nickel sample 50 mixed in a quartz tube 40 which is provided in the tubular reactor 20, and a reaction furnace 30 for allowing the tubular reactor 20 provided with the sample 50 to obtain sufficient adiabatic reaction. It further includes an igniter 81 for igniting the sample 50 by current of a high voltage supplied from a transformer 80 if the tubular reactor 20 reaches an adiabatic reaction temperature by the reaction furnace 30, an X-Y register 60 for registering a mixed powder molding reaction temperature of the sample 50 ignited by the igniter 81, the mixed powder molding reaction temperature being supplied from a sample temperature sensing thermo couple 61, a reaction furnace controlling thermo couple 71 for sensing heat supplied to the heating element 31 of the reaction furnace 30, and a temperature controller 70 for controlling a temperature of the sensed heat.

The gas flow controller 11 is mounted in the inert gas receptacle 10 and controls inner pressure of the tubular reactor 20 at 1~2 Torr.

The sample is preheated within the tubular reactor 22 at a temperature of 250°C ~ 550°C to obtain sufficient adiabatic reaction because the sample has a low adiabatic temperature during rotational synthesis reaction of titanium and nickel. If the sample 50 preheated within the tubular reactor 20 reaches a predetermined temperature, the sample 50 is ignited by the igniter 81. That is, the sample 50 acts as a mixed powder molding body ignited by the igniter 80.

Furthermore, an end portion of the sample 50 may partially be removed. A mixed powder of titanium and boron(B) may be filled in the portion where the sample 50 is partially removed, so as to be used as an auxiliary means such as a chemical furnace.

In the present invention as described above, two or more elements are reacted to form a compound, and a powder is manufactured based on heat generated when forming the compound. Also, the artificial medulla is manufactured by a hot rotational synthesis method. In the hot rotational synthesis method, when manufacturing a metal compound, impurities mixed with the raw powders in the synthesis reaction of high temperature are removed. Thus, a compound of high purity can be obtained. In more

detail, the method for manufacturing an artificial porous titanium nickel medulla based on the hot rotational synthesis method includes the steps of drying raw powders of titanium and nickel under the vacuum state to remove
5 moisture and surface absorption materials, thereby minimizing an amount of gas generated during synthesis reaction, dry-mixing the raw powders dried during the drying step with each other at a ratio of about 1:1 in atomic amount to manufacture mixed powders having uniform
10 compositions, molding the mixed powders within a cylindrical quartz tube with compression or without pressure depending on a desired porosity and pore size, reacting the mixed powders molded in the molding step in a reaction furnace by a hot rotational synthesis method,
15 cooling titanium-nickel products reacted in the reacting step using a reservoir for a cooling liquid, and removing impurities on a surface of the cooled titanium-nickel products to process the titanium-nickel products in a desired shape.

20 Meanwhile, in the drying step in which raw powders of titanium and nickel are dried under the vacuum state to remove moisture, thereby minimizing an amount of gas generated during synthesis reaction, the inert gas contained in the inert gas receptacle is controlled and
25 supplied by the gas flow controller 11 to maintain the inner pressure of the tubular reactor 20 provided with

titanium and nickel powders at 1 ~ 2 Torr. The reaction furnace controlling thermo couple 71 senses an inner temperature of the tubular reactor 20. If the inner temperature of the tubular reactor 20 is 250°C ~ 550°C or
5 below, the temperature controller is operated to supply insufficient heat from the heating element 31 provided in the reaction furnace 30. If the inner temperature of the tubular reactor 20 reaches 250°C ~ 550°C by means of the heating element 31, the sample 50 in the quartz tube 40
10 is ignited by the igniter 81 which receives high power from the transformer 80.

As described above, in a state that the sample 50 is ignited, the ignition wave is propagated into the mixed powder molding body to complete reaction. Then, in
15 a state that the inert gas is continuously supplied at high pressure, the tubular reactor 20 is extracted from the reaction furnace 30. The mixed powder molding body is then cooled by a reservoir for a cooling liquid (not shown).

20 Surface impurities of the cooled mixed powder molding body, i.e., cooled titanium-nickel products, are removed by grinding or turning so that the cooled titanium-nickel products are processed by the discharge processing method in a desired shape. It is noted that
25 the surface impurities are also pickled with a mixed

solution of distilled water of hydrofluoric acid and nitric acid, so as to be removed.

Industrial Applicability

5 The aforementioned apparatus and method for manufacturing an artificial porous titanium nickel medulla by using a hot rotational synthesis method has the following advantages.

 When manufacturing the artificial medulla using a
10 titanium nickel material having shape memory effect and super elastic effect, a porous structure is controlled to obtain mechanical qualities required for an implant material at a site of a shattered medulla which absolutely requires initial stability, thereby enhancing
15 osteoconduction effect and bone ingrowth, and a newly grown medulla is created after implant and then its material qualities are controlled to be similar to a human medulla, thereby obtaining biocompatibility. Thus, it is not subject to various limitations as to whether
20 biocompatibility exists or not depending on both the site of the shattered medulla and body qualities of the patient.

 The foregoing embodiments are merely exemplary and are not to be construed as limiting the present invention.
25 The present teachings can be readily applied to other types of apparatuses. The description of the present

invention is intended to be illustrative, and not to limit the scope of the claims. Many alternatives, modifications, and variations will be apparent to those skilled in the art.

CLAIMS

1. A method for manufacturing an artificial porous titanium nickel medulla by using a hot rotational

5 synthesis method comprising the steps of:

drying raw powders of Ti and Ni under the vacuum state to remove moisture and surface absorption materials, thereby minimizing an amount of gas generated during synthesis reaction;

10 dry-mixing the raw powders dried during the drying step with each other at a ratio of about 1:1 in atomic amount to manufacture mixed powders having uniform compositions;

molding the mixed powders within a cylindrical quartz tube with compression or without pressure
15 depending on a desired porosity and pore size;

reacting the mixed powders molded in the molding step in a reaction furnace by a hot rotational synthesis method;

20 cooling titanium-nickel products reacted in the reacting step using a reservoir for a cooling liquid; and

removing impurities on a surface of the cooled titanium-nickel products to process the titanium-nickel products in a desired shape.

25

2. The method according to claim 1, wherein a

molding body molded within the quartz tube has a diameter of 20mm or greater to avoid instability (extinguishment) of the ignition wave due to heat loss and to obtain adiabatic reaction conditions.

5

3. The method according to claim 1, wherein the molding body has a porosity of 30 ~ 70%.

4. The method according to claim 1, wherein surface
10 impurities of the cooled titanium nickel products are removed by grinding or turning so that the cooled titanium-nickel products are processed by a discharge processing method in a desired shape, and the surface impurities are also pickled with a mixed solution of
15 distilled water of hydrofluoric acid and nitric acid, so as to be removed.

5. An apparatus for manufacturing an artificial porous titanium nickel medulla by using a hot rotational
20 synthesis method comprising:

an inert gas receptacle (10) for containing an inert gas of which amount is controlled by a gas flow controller (11);

a tubular reactor (20) to which the inert gas
25 contained in the inert gas receptacle (10) is supplied;

a titanium-nickel sample (50) mixed in a quartz

tube (40) which is provided in the tubular reactor (20);

a reaction furnace (30) for allowing the tubular reactor (20) provided with the sample (50) to obtain sufficient adiabatic reaction;

5 an igniter (81) for igniting the sample (50) by current of a high voltage supplied from a transformer (80) if the tubular reactor (20) reaches an adiabatic reaction temperature by the reaction furnace (30);

10 an X-Y register (60) for registering a mixed powder molding reaction temperature of the sample (50) ignited by the igniter (81), the mixed powder molding reaction temperature being supplied from a sample temperature sensing thermo couple (61);

15 a reaction furnace controlling thermo couple (71) for sensing heat supplied to the heating element (31) of the reaction furnace (30); and

a temperature controller (70) for controlling a temperature of the sensed heat.

20 6. The apparatus according to claim 5, wherein the gas flow controller (11) is mounted in the inert gas receptacle (10) and controls an inner pressure of the tubular reactor (20) at 1~2 Torr.

25 7. The apparatus according to claim 5, wherein the sample (50) is preheated within the tubular reactor (22)

at a temperature of 250°C ~ 550°C to obtain sufficient
adiabatic reaction because the sample has a low adiabatic
temperature during rotational synthesis reaction of
titanium and nickel, and if the sample (50) preheated
5 within the tubular reactor (20) reaches a predetermined
temperature, the sample (50) is ignited by the igniter
(81).

8. The apparatus according to claim 5, wherein an
10 end portion of the sample (50) may partially be removed,
and a mixed powder of titanium and boron (B) may be
filled in the portion where the sample 50 is partially
removed.

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FIG. 1

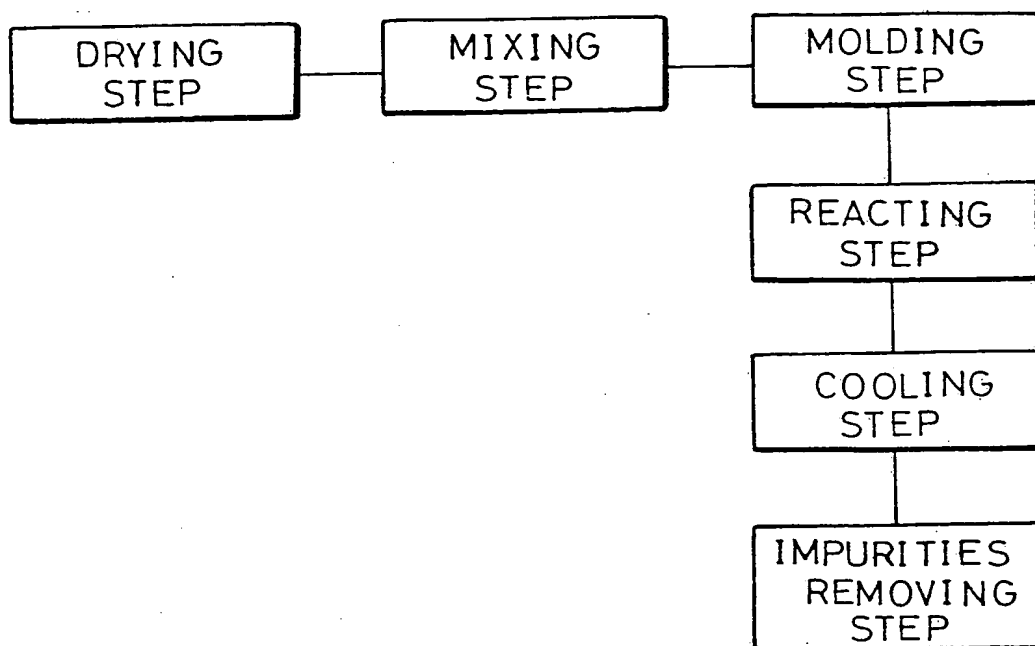
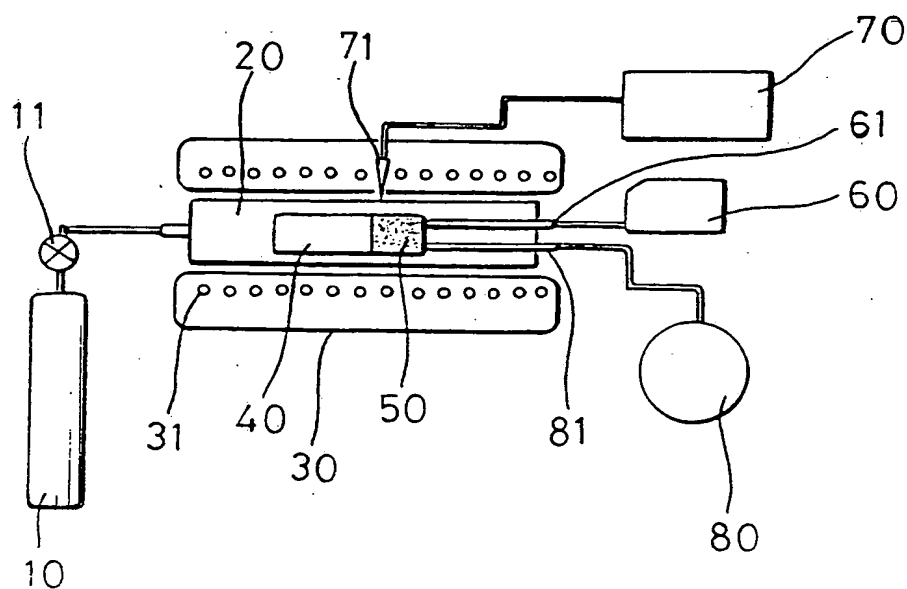


FIG. 2



INTERNATIONAL SEARCH REPORT

International application No.
PCT/KR 00/00938

CLASSIFICATION OF SUBJECT MATTER

IPC⁷: A61L 27/06

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC⁷: A61L 27/00; C22C 14/00; C22C 19/00

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPODOC, PAJ, WPI

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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X	SU 662270 A (ITIN V.I.) 15 May 1979 (15.05.79) (abstract) World Patent Index [online] [retrieved on 14 November 2000 (14.11.00)]. Retrieved from EPO WPI Database, DW 198004, Accession No. 1980-07030C.	1,3
A	DE 4210801 A1 (ROSSAR INGENEERING GbR. Gesellschaft für Forschung, Technologie und Patentverwertung) 5 November 1992 (05.11.92) pages 2-4.	1,3
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☒ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

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„Y“ document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

„&“ document member of the same patent family

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INTERNATIONAL SEARCH REPORT

International application No.

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C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 52-050909 A (NATIONAL RES INST M) 23 April 1997 (23.04.97) (abstract) World Patent Index [online] [retrieved on 13 November 2000 (13.11.00)]. Retrieved from EPO WPI Database, DW 197722, Accession No. 1977-39097Y.	1
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